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21) International Application Number: PCT/E. 22) International Filing Date: 9 March 1993 30) Priority data: 92302334.5 18 March 1992 (18.03.92) (34) Countries for which the regional or international application was filed: 1) Applicant (for GB IE only): UNILEVER PLC Unilever House, Blackfriars, London EC4P 4I 1) Applicant (for all designated States except GB IE ILEVER N.V. [NL/NL]; Weena 455, P.O. Box 3000 DK Rotterdam (NL).	(09.03.9 AT et [GB/GH BQ (GB)	 (75) Inventors/Applicants (for US only): NAKONECZNYI, Anna [GB/GB]; 363 London Road, Bedford MK42 0PX (GB). SMITH, Kevin, Warren [GB/GB]; 16 Arundel Drive, Bedford MK41 8NP (GB). (74) Agent: JOPPE, Hermina, L., P.; Unilever N.V., Patent Division, P.O. Box 137, NL-3130 AC Vlaardingen (NL). (81) Designated States: US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, NL, PT, SE). Published

(57) Abstract

A method of preparing a fractionated polyol fatty acid polyester, whereby a polyol fatty acid polyester, obtained by esterifying a polyol with two or more fatty acids or derivatives thereof, is subjected to fractionation. Also the invention relates to a food product comprising from 1-100 % by weight of fatty substances, wherein from 50-100 % of the fatty substances are fractionated polyol fatty acid polyesters.

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EDIBLE FAT-CONTAINING PRODUCTS

The present invention relates to edible fat-containing products comprising an indigestible fat-replacer.

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In this specification, unless otherwise indicated, the term 'fat' refers to edible fatty substances in a general sense, including natural or synthesized fats and oils consisting essentially of triglycerides such as, for example, soybean oil, sunflower oil, palm oil, coconut oil, fish oil, lard and tallow, which may have been partially or completely hydrogenated or modified otherwise, as well as non-toxic fatty materials having properties similar to triglycerides, herein referred to as fat-replacers, which materials may be partially or fully indigestible. The terms fat and oil are used interchangeably.

Over the last decade many non-triglyceride fatty substances have been described as potential fat-replacers in food products. Examples thereof are waxes, e.g. jojoba oil and hydrogenated jojoba oil, polysiloxanes, acylated glycerides, polyalkoxyglycerolethers, dicarboxylic acid esters, polyol fatty acid polyesters and the epoxy extended derivatives thereof. Examples of disclosures of fat-replacers are e.g. DD 207 070, Journal of Food Science 49, 419-428 (1984), US 3,600,186, US 4,005,195, US 4,005,196, US 4,034,083, US 4,582,715, US 4,582,927, EP 0 233 856, EP 0 236 288, EP 0 235 836 and EP 0 254 547.

In particular polyol fatty acid polyesters, and more specifically the sugar fatty acid polyesters, such as e.g. the sucrose fatty acid polyesters, are receiving increased attention as low-calorie fat-replacers in edible products. Substantially indigestible for human beings they have physical and organoleptic properties very similar to triglyceride oils and fats conventionally used in edible products.

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Polyol fatty acid polyesters are also reported to have use as pharmaceutical agents e.g. in view of their ability to take up fat-soluble substances, such as in particular cholesterol, in the gastro-intestinal tract, and subsequently remove those substances from the human body.

Suitable polyol fatty acid polyesters are derived from aliphatic or aromatic polyols which comprise at least four free hydroxyl groups. Such polyols in particular include the group of sugar polyols, which comprises the sugars, i.e. the mono-, di- and polysaccharides, the corresponding sugar alcohols and the derivatives thereof having at least four free hydroxyl groups. Examples of sugar polyols include glucose, mannose, galactose, xylose, fructose, sorbose, tagatose, ribulose, xylulose, maltose, lactose, cellobiose, raffinose, sucrose, erythritol, mannitol, lactitol, sorbitol, xylitol and alpha-methylglucoside. A particularly preferred polyol is sucrose.

The term 'polyol fatty acid polyester' is intended to refer to any such polyesters or mixtures thereof which have a degree of conversion of 70 % or more, i.e. of which, on an average, 70 % or more of the polyol hydroxyl groups have been esterified with fatty acids. Preferred polyol fatty acid polyesters for use in the present invention have degrees of conversion of 85 % or more, or even 95 % or more.

The fatty acid residues in the polyol fatty acid polyesters

may be derived from naturally occurring or synthetic fatty
acids per se, or suitable sources thereof, such as natural
triglyceride fats and oils or their corresponding loweralkyl esters. The fatty acids may be saturated or
unsaturated, branched or straight fatty acids containing

from 8 to 24 carbon atoms, in particular 12 to 18 carbon
atoms, such as lauric, myristic, palmitic, stearic, oleic,
elaidic, and linoleic acids. Suitable natural sources are

the vegetable oils, such as sunflower, safflower, rapeseed, cottonseed, palm kernel, palm and soybean oils. Also other oils, for example marine oil or animal oil may be used. If so required, conventional techniques may be used to first introduce the necessary degree of saturation. Suitable such techniques include full or partial hydrogenation, interesterification, and fractionation, and may be used before or after conversion to the polyol fatty acid polyesters.

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A problem with polyol fatty acid esters obtained by conventional techniques is that the mouthfeel, crystallisation and melting properties are often not optimal. This problem is especially apparent in polyol fatty acids which are intended to be used in confectionery products such as chocolate.

Confectionery chocolate products preferably are solid and crisp at ambient temperature, and at the same time fluid and soft at body temperature. For combining these two desired properties a steep melting profile between 20 °C and 35 °C, especially between 30°C and 35° is important.

EP 350 981 (Unilever) describes polyol fatty acid polyesters for use in confectionery products, said polyesters having an N₂₀ of 50-100 and an N₃₇ of 0-5.

EP 271 951 (P&G) describes cocoa butter substitutes comprising sucrose fatty acid esters which are solid at ambient temperature and which have a lower solid fat content at body temperature.

US 4,810,516 (P&G) describes reduced calorie chocolate confection compositions comprising polyol fatty acid

35 polyesters having a clear melting point of 30-36 °C and an SCI of at least 66 at a temperature of 6.6 °C below its clear melting point.

A problem with the polyol fatty acid polyesters of the above documents is that for obtaining the desired melting properties it is often necessary to specifically select the source of fatty acids for preparing the polyol fatty acid polyester. This relatively narrow choice of fatty acid materials may provide a cost increase and furthermore may render the industrial production of the polyol fatty acid polyesters too much dependant on one or more supplying sources. Another problem with the polyol fatty acid polyesters as disclosed in these documents is that they often tend to undergo further crystallisation after preparation, such that although the initial N₃₅ is relatively low, this value increases in time providing an undesired waxiness and unsatisfactory mouthfeel to the product.

The melting porperties of fats can suitably be determined by measuring the N-line. The N-line is the graph of N_t-values versus the temperature t. The N_t-value is

conveniently measured by the nuclear magnetic relaxation technique and is a direct measure of the level of solid fat content at temperature t. This method is suitably described in Fette, Seifen, Anstrichmittel 80(5), 180-186 (1978). To some extent the measurement of N_t-values is dependent on the temperature profile used to prepare the samples for the NMR-measurement. For the purposes of the present invention the following preparatory temperature profile is adopted:

30 minutes at 60°C, 90 minutes at 0°C, 40 hours at 26°C, again 90 minutes at 0°C and finally 60 minutes at the temperature of the measurement, after which the NMR-measurement is carried out.

It is an object of the present invention to provide polyol fatty acid polyesters with a tailored melting behaviour

35 which can be made from a variety of fatty acid sources.

Surprisingly it has been found that the melting properties

of polyol fatty acid polyesters can suitably be influenced by fractionating polyol fatty acid polyesters. In particular it has been found that by applying suitable fractionation techniques some parts of the N-line can be 5 made relatively lower while other parts remain the same or even increase in N-value. For example by using fractionation of polyol fatty acid polyesters it is possible to have a significant relative decrease of the Nvalue at 35 °C, while the N-value at 20 °C shows a small 10 relative decrease or even an increase. This renders the relevant fraction of the polyol fatty acid polyester more suitable for use in chocolate confectionery products than the unfractionated polyol fatty acid polyesters. Similar advantages may apply to the use of fractionated polyol 15 fatty acid polyesters in other food products, for example spreads, (savoury) snacks, bakery products, fried food products, cooking fats etc.

If for example for polyol fatty acid esters for use in chocolate confectioneries the N-value at 20°C has to increase, this may be done by removing the soft fraction of the polyester. If, on the other hand the N-value at 35°C has to decrease, this can be done by removing the hard fraction of the polyol fatty acid polyester by fractionation. For combining the two effects, both fractions may be removed.

By-products from the fractionation i.e. fractions of the polyol fatty acid esters which are left over after

30 fractionation may advantageously be used for other purposes e.g. hard fractions for hardstocks in spreads and margarines and soft fractions for frying oils, soft confectionery filling fats etc.

Accordingly the present invention relates to a method of preparing a fractionated polyol fatty acid polyester, whereby a polyol fatty acid polyester, obtained by

esterifying a polyol with two or more fatty acids or derivatives thereof, is subjected to fractionation. Also the invention relates to fractionated spe's obtained by this method and to food products comprising from 1-100% by weight of polyol fatty acid polyesters, wherein from 50-100% of the polyol fatty acid polyesters are fractionated polyol fatty acid polyesters.

For the purpose of the invention it is important to realise

that the term fractionation does not include any washing
steps etc. which may be carried out during the preparation
of the polyol fatty acid polyesters. In this specification
fractionation refers to a separate treatment step to be
applied to polyol fatty acid polyesters after their

preparation.

As indicated above any polyol fatty acid polyester obtained by conventional preparation methods may advantageously be subjected to fractionation to obtain the desired changes in melting behaviour. Suitable methods for obtaining non-fractionated polyol fatty acid polyesters are for example disclosed in EP 132,941, EP 132,293, US 3,963,699, EP 256,585, EP 301,634, EP 319,091 and EP 322,971. A particular preferred process is described in and involves the steps as disclosed in EP 256,585.

In a preferred embodiment of the invention, nonfractionated polyol fatty acid polyesters are used which
contain at least two different fatty acid residues,

sepecially preferred are polyol fatty acid polyesters
wherein the most abundant fatty acid residue is present in
an amount of from 5 to 95% by weight of the fatty acid
residues. In a particular preferred embodiment of the
invention the non-fractionated polyol fatty acid residue is
obtained from naturally occurring fatty acid sources or
mixtures thereof. Especially advantageous is the use of the
following fatty acid sources: palm-kernel oil, palm oil,

soybean oil, low erucic rape seed oil, coconut oil, sheanut oil, sunflower oil, cotton seed oil and mixtures thereof, a particular advantageous fatty acid source includes a mixture of palm kernel and palm oil in a weight ratio of 10 : 1 to 1 : 10. When desired the fatty sources may be partially or fully hydrogenated prior to usage. Preferably the octa ester content of non-fractionated polyol fatty acid esters is more than 80%, more preferred more than 85%, most preferred more than 90%. Also preferably sucrose is used as the polyol.

Although applicants do by no means want to be bound by this theory, it is believed that the improved melting properties of fractionated polyol fatty acid polyesters are due to the specific division of fatty acid residues over the fractionated polyol fatty acid ester molecules. For example if the hard fraction is removed, this generally results in the removal of those polyesters containing high proportions of longer chain (e.g. C₁₆ or C₁₈) saturated fatty acids while maintaining polyesters with a lower proportions of these fatty acids.

If the soft fraction is removed, those polyesters are removed which contain a high proportion of shorter chain and unsaturated fatty acids (e.g. C₈, C₁₀ and C_{18:1}) as the softer fraction.

Irrespective of which fraction is removed, the result will be that the fractionated polyol fatty acid polyester will no longer have a random distribution of fatty acids over the polyol. This non-randomised nature of fractionated polyol fatty acid esters may be detected by measuring the N-line of the fractionated material and comparing it to the N-line of a polyol fatty acid polyester with the same fatty acid composition, but prepared by random esterification and/or by comparing the clear point of the two materials.

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Preferably the N-line of the fractionated material satisfies one or more of the following criteria when compared to the random polyester material:

- a) the clear point of the fractionated polyol fatty acid material is at least 0.5°C lower than that of the randomised material, more preferably the reduction in clear point is from 1-10°C, most preferred 2-5°C;
- b) the N-value at 20°C is at least 5% higher for the fractionated material than for the randomised material, more preferred at least 10%, most preferred at least 15%.

Further tests e.g. for distinguishing between fractionated 15 polyol fatty acid polyesters and mixtures of random material may involve SFC, GC, DSC, HPLC, selective absorption techniques, NMR and/or IR.

polyesters, conventional wet and dry fractionating techniques may be used. Preferably the fractionation takes place at a temperature of between -20°C and 20°C, more preferred -5 to 10°C, most preferred from -3 to 4°C. Higher fractionation temperatures have the advantage of resulting in higher yields and hence less raw material losses. If wet fractionation is applied the preferred solvents are relatively non-polar, like hexane and isopropyl alcohol. Dry fractionation is preferably carried out by cooling/winterising followed by filtering or oil pressing. Suitable fractionation techniques are for example disclosed in EP 424,997 and functional properties of fats in foods, J. Solms, 193-201.

Food compositions in accordance to the invention comprise polyol fatty acid polyesters as part or all of the fatty substances. Apart from polyol fatty acid polyesters as defined herein, food compositions of the invention may

comprise digestible fatty substances such as triglyceride materials and non-glyceride fatty substances.

The level of these fatty substances other than the polyol fatty acid polyesters may be up to 90 wt%, based on the total weight of the fatty substances, preferably, however the level is from 0-40 wt%, more preferred 0-10 wt%, most preferred food compositions of the invention are substantially free from fatty substances other than polyol fatty acid polyesters.

Depending on the type of food composition, the total level of fatty substances in the edible composition of the invention may be from 1-100 wt%, for example 5-95 wt%. Some edible compositions like shortenings, chocolate and dressings may comprise relatively high levels of fatty substances, for example 30-90 wt%. Other edible compositions like sauces, soups, bakery products etc may comprise lower levels of fatty substances for example 1-30 wt%.

In any case, the fatty substances which are present in the food compositions of the invention comprise a fractionated polyol fatty acid polyester material, the level of fractionated polyol fatty acid polyesters preferably being from 50-100 wt%, more preferred 70-97 wt%, most preferred 80-95 wt% based on the total weight of fatty substances in the food product.

The optional conventional fat-component may be triglyceride oils or fats of animal or vegetable origin. Suitable conventional triglyceride fats and oils include, optionally partially or fully hydrogenated, coconut oil, palmkernel oil, palm oil, marine oils, lard, tallow fat, butter fat, cocoa butter fat, soybean oil, safflower oil, cotton seed oil, rapeseed oil, corn oil sunflower oil and mixtures thereof.

In terms of rheology the selection of the appropriate polyol fatty acid polyesters or mixture of polyesters and conventional fat materials strongly depends upon the particular application envisaged, and may range of polyol fatty acid polyester having a low melting rheology, in products where this is preferred or to more viscous or even solid rheology in compositions having a more structured fat phase such as margarines, spreads, shortenings and confectionery products.

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The present invention is not specific to edible fatcontaining compositions in any particular food area. It may
be suitably be applied to food products such as spreads,
margarines, creams, shortenings, bakery products such as
doughs, cakes and biscuits, fried and snack products,
fresh, hard and processed cheeses, meat emulsions,
mayonnaise and dressings, confectionery products, such as
desserts, fillings, chocolates, candies, chews, and icecreams.

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Particularly preferred however is the use of fractionated polyol fatty acid polyesters in chocolate confectionery products. Suitable chocolate confectionery products preferably comprise from 25-60% by weight of fatty

25 substances, 0.001 to 60% by weight of sugar or other sweeteners such as for example aspartame, 5-35% by weight of cocoa powder or chocolate liquor and 0-40% of milk solids, 0-5% flavouring and/or colouring materials, 0-5% lecithin or other emulsifying agent and up to 100% of other suitable materials, e.g. carbohydrate or other bulking agents.

In another preferred embodiment of the invention the chocolate confectionery product of the invention comprises fatty substances having an N-value at 20 °C of at least 60, more preferred 70-100, most preferred 80-95 and an N-value at 35°C of less than 15, more preferred from 0-8, most

preferred from 0-3. Preferably at least 50% by weight of these fatty substances are fractionated polyol fatty acid polyester materials, more preferred 70-100%, most preferred 90-100%.

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In a particular preferred embodiment of the invention a fractionated polyol fatty acid is obtained by fractionating a polyol fatty acid polyester obtained from palm kernel and palm oil fatty acid residues in a weight ratio of 10 : 1 to 1 :10 such that the N₃₅ is less than 3 more preferred less than 2, most preferred less than 1 and the N₂₀ is above 60 more preferred more than 80, most preferred more than 90. This fractionated polyol fatty acid residue is then preferably used as the predominant fatty substance in chocolate confectionary products at a level of 25 to 45%.

Preferably the above mentioned N-values can be observed, both directly after preparation and after storage for say 1 month at ambient temperature.

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Chocolate confectionery products of the invention can be made by conventional techniques, such as for example described in US 4,810,516. Preferably the cooling regime applied to the chocolate is such that the product can be demoulded within 30 minutes after moulding.

The invention will be further illustrated by means of the following example:

Example I

A sucrose fatty acid polyester is obtained from a 62: 38 mixture of palm kernel oil hardened to a slip melting point of 39 °C and a palm oil hardened to a slip melting point of 58 °C, which is converted to the respective methyl esters and then reacted with sucrose in the presence of a potassium carbonate catalyst.

The non-fractionated sucrose polyester was fractionated in 10 hexane (weight ratio solvent to fat 2:1) at a number of temperatures. The N-values of the obtained fractionated sucrose polyesters were measured as described hereinabove at various temperatures. The following results were obtained:

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Example II table 1

	fractionation						
	TEMP	N20	N25	N30	N32.5	N35	N40
20							
	2°C	81.5	70.9	55.0	37.8	11.5	0
	1°C	80.2	68.0	49.8	31.4	0	0
	0°C	78.1	65.1	45.2	25.8	0	0
	-2°C	74.9	52.5	27.7	1.7	0	0
25	-5°C	68.6	39.5	13.0	0.4	0	0
	non-						
	fractionated	81.5	72.0	58.4	40.1	15.2	0.3

A plain chocolate is prepared from the above fractions and non-fractionated materials according to the following composition:

	<u>ingredient</u>	wt %
	polyol fatty acid polyester	32 %
35	sugar	49.6 %
	cocoa powder	18 %
	lecithin	0.4 %

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The ingredients were mixed at 50°C followed by milling at 25°C using a triple roller mill, conched for 5 hours at 50°C in an end-runner, conch moulded, cooled at 10°C for 30 minutes, demoulded and stored at ambient temperature for 1 month.

The mouthfeel of the non-fractionated polyester containing chocolate is inferior to the mouthfeel of the other

10 chocolates. Especially the chocolate made from the fraction at 1°C has an excellent mouthfeel and melting properties.

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Claims

- 1. A method of preparing a fractionated polyol fatty acid polyester, whereby a polyol fatty acid polyester, obtained by esterifying a polyol with fatty acids or derivatives thereof, is subjected to fractionation.
- 2. A method according to claim 1, wherein the polyol fatty acid polyester before fractionation contains at least two different fatty acid residues.
- 3. A method according to claim 1 or 2, wherein the polyol fatty acid polyester before fractionation contains fatty acid residues of palm kernel and palm oil in a weight ratio of 10:1 to 1:10.
- 4. A method according to claims 1-3, wherein the fractionation takes place at a temperature between -20° and 20°C.
- 5. A method according to claims 1-4, wherein wet fractionation is applied while using a non-polar solvent preferably hexane or isopropyl alcohol.
- 6. Fractionated polyol fatty acid polyesters obtained by a method of claims 1-5.
- 7. Fractionated polyol fatty acid polyesters wherein the fractionated material when compared to a random polyol fatty acid polyester of the same fatty acid composition, satisfies one or both of the criteria
 - a) the clear point of the fractionated polyol fatty acid material is at least 0.5°C lower than that of the randomised material:

- b) the N-value of the fractionated polyol fatty acid material at 20°C is at least 5% higher than the N-value at 20°C of the randomised material.
- 8. Food products comprising from 1-100% by weight of fatty substances, wherein from 50-100 wt% of the fatty substances are fractionated polyol fatty acid polyesters in accordance to claim 6 or 7.
- 9. Food product according to claim 8 being a chocolate confectionery product.
- 10. Food product according to claim 8 or 9 comprising
 - a) 25-60 wt% of fatty substances;
 - b) 0.001-60 wt% of sugar or other sweeteners;
 - c) 5-35 wt% of cocoa powder or chocolate liquor;
 - d) 0-40 wt% of milk solids;
 - e) 0-5 wt% of flavouring and/or colouring materials;
 - f) 0-5 wt% of emulsifying agents.

International Application No

L CLASSIFIC	ATION OF SUBJ	ECT MATTER (if several classification s	symbols apply, indicate all) ⁶	
According to		Classification (IPC) or to both National C	Cassification and IPC	23D9/00
II. FIELDS SI	EARCHED			
		Minimum Docum	entation Searched?	
Classification	System		Classification Symbols	
Int.C1.	5	CO7H ; C11B ;	A23G ; A23D	
			than Minimum Documentation are Included in the Fields Searched ⁸	
III DOCUME	NTE CONSIDERE	D TO BE RELEVANT 9		
Category °		cument, 11 with indication, where appropri		7.1
Category	Citation of De	cument, with indication, where appropri	ate, of the relevant passages "	Relevant to Claim No.13
Υ	2 May 19	424 997 (UNILEVER NV) 991 Whole document		1-10
Y	EP,A,0 3 25 July	 378 876 (UNILEVER NV)	claim 1:	1-10
Υ	example			1-10
	11 July see page		- page 4,	1-10
Y	NESTLE S 14 Janua see page	ary 1987 e 1, line 40 - line 50;		1-10
	examples	5	•	
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IV. CERTIFIC	ATION			·
Date of the Act	tual Completion of t	he International Search	Date of Mailing of this International Ser	arch Report
T		JLY 1993	2 7, 07, 93	
international S	eurching Authority EUROPEA	IN PATENT OFFICE	Signature of Authorized Officer DAY G.J.	

International Application No

III. DOCUM	ENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)	
Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
	J. SOLMS 'Fette als funktionelle Bestandteile von Lebensmitteln' 1973 , FORSTER-VERLAG AG , ZURICH CH	. 1
	see page 193 - page 201	
-		

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

ΕP 9300551 SA 70936

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82